Exhibit U

PATENT
ATTORNEY DOCKET NO.: 056100-5078

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Reissue Application of:	)	
	)	
Patentees: Robert H. FRUSHOUR et al.	)	
	)	
U.S. Patent No. 6,811,610 B2	)	Group Art Unit: unassigned
	)	
Issued: November 2, 2004	)	Examiner: unassigned
	)	
Reissue Application No.: unassigned)	)	
	)	
For: METHOD OF MAKING ENHANCED CVD DIAMOND	)	

ASSENT OF ASSIGNEE TO REISSUE APPLICATION

Commissioner for Patents
U.S. Patent and Trademark Office
Mail Stop REISSUE
U.S. Patent and Trademark Office
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

The undersigned, assignee of the entire right, title, and interest in the above-mentioned letters patent, hereby assents to the accompanying reissue application.

CERTIFICATE UNDER 37 C.F.R. 3.73(b)

Carnegie Institution of Washington ("Carnegie") is the assignee of the entire right, title, and interest in and to the invention and patent identified above by virtue of Assignment from Diamond Innovations, Inc. recorded on December 31, 2008 at Reel 022043, Frame 0167. I have reviewed the Assignments of record that grant the entire right, title, and interest in the above-identified patent to Carnegie subject to a license granted to Diamond Innovations and, to the best of my knowledge and belief, title to the patent is in

Carnegie. A copy of the Assignment is attached as **Exhibit A**. Also attached as **Exhibit B** is a copy of the ownership record for the patent as obtained from the publicly available records.

The undersigned (whose title is supplied below) is empowered to sign this certificate on behalf of the Assignee.

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements are made with the knowledge that willful false statements, and the like so made, are punishable by fine or imprisonment, or both, under Section 1001, Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

Carnegie Institution of Washington

By: _____

Gary Kowalczyk
Director of Administration and Finance

Date: 1/15/2009

EXHIBIT A

APPENDIX A
CONFIRMATION OF ASSIGNMENT

WHEREAS, Diamond Innovations, Inc., a Delaware corporation having a primary address of 6325 Huntley Road, Worthington, OH 43082 (the "ASSIGNOR") is the owner of record of United States Patent No. 6,811,610, entitled "Method of Making Enhanced CVD Diamond" (the "PATENT"); and

WHEREAS, Carnegie Institution of Washington, having an address of 1530 P Street NW, Washington, DC 20005 ("ASSIGNEE"), desires to acquire, subject to a license to ASSIGNOR, the PATENT.

NOW, THEREFORE, for good and valuable consideration, the receipt for and sufficiency of which is hereby acknowledged, ASSIGNOR does hereby sell, assign, transfer and set over unto ASSIGNEE, its legal representatives, successors, and assigns, its entire right, title and interest in and to the PATENT, subject to the license granted to ASSIGNOR pursuant to that certain Assignment and Non-Exclusive License Agreement between ASSIGNOR and ASSIGNEE, dated as of even date herewith.

IN WITNESS WHEREOF, the parties hereto have caused this ASSIGNMENT to be duly executed and delivered as of June 30, 2008.

Carnegie Institution of Washington

Signature
GARY KOWALCZYK
Printed Name
DIRECTOR, ADMINISTRATION AND FINANCE
Title

Diamond Innovations, Inc.

Signature
TANYA D. FRATTO
Printed Name
PRESIDENT & CEO
Title
9/24/08

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of	Wei LI	Confirmation No.	1281
Serial No.:	12/362,529	Art Unit:	
Filed:	January 30, 2009	Examiner:	
For:	**METHOD OF MAKING ENHANCED CVD DIAMOND**	Atty. Docket No:	056100-5078-US

RESPONSE TO NOTICE OF MISSING PARTS OF REISSUE APPLICATION

U.S. Patent and Trademark Office
Randolph Building
401 Dulany Street
Alexandria, Virginia 22314 – **MAIL STOP MISSING PARTS**

Sir:

In response to the Notice to File Missing Parts of Reissue Application mailed on February 24, 2009, attached is the reissue specification in double-column format as required by 37 CFR 1.173(a)(1).

A fee of **$130.00** is due for the submission of this Response. Please charge the required fee to **Morgan, Lewis & Bockius LLP** Deposit Account No. 50-0310.

Date: March 3, 2009

Respectfully submitted,

Paul N. Kokulis Reg.. No. 16,773

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US006811610B2

(12) **United States Patent**
Frushour et al.

(10) **Patent No.: US 6,811,610 B2**
(45) **Date of Patent: Nov. 2, 2004**

(54) **METHOD OF MAKING ENHANCED CVD DIAMOND**

(75) Inventors: **Robert H. Frushour**, Ann Arbor, MI (US); **Wei Li**, Ann Arbor, MI (US)

(73) Assignee: **Diamond Innovations, Inc.**, Worthington, OH (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 39 days.

(21) Appl. No.: **10/161,266**

(22) Filed: **Jun. 3, 2002**

(65) **Prior Publication Data**

US 2003/0230232 A1 Dec. 18, 2003

(51) **Int. Cl.**[7] C23C 16/27

(52) **U.S. Cl.** 117/88; 427/249.7; 427/249.8; 427/370; 427/372.2; 427/444; 427/902

(58) **Field of Search** 427/249.7, 249.8, 427/370, 372.2, 444, 902; 117/88

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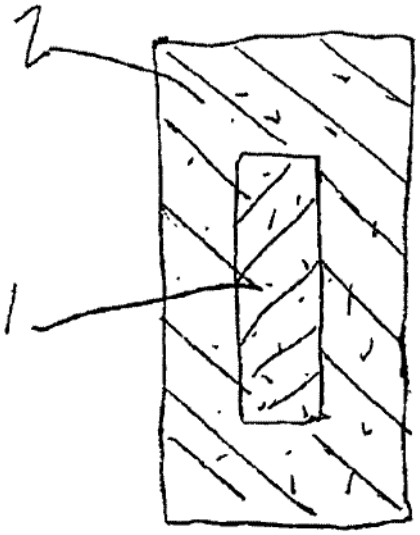
Primary Examiner—Timothy Meeks
(74) *Attorney, Agent, or Firm*—Pepper Hamilton LLP

(57) **ABSTRACT**

Single crystal CVD diamond is heated to temperatures of 1500° C. to 2900° C. under a pressure that prevents significant graphitization. The result is a CVD diamond with improved optical properties.

7 Claims, 1 Drawing Sheet



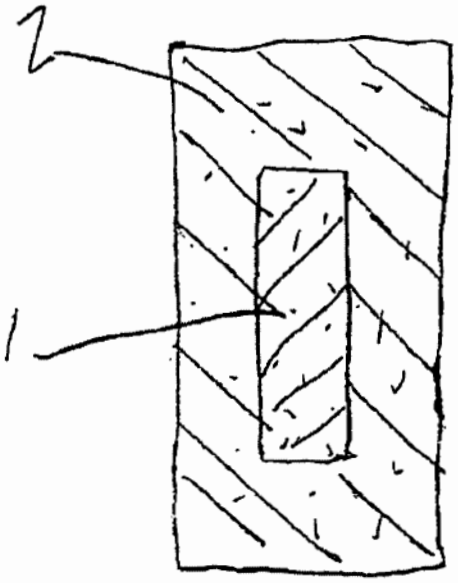


FIG. 1

METHOD OF MAKING ENHANCED CVD DIAMOND

BACKGROUND

This invention relates to a method of improving the optical, electrical, thermal, and mechanical properties of chemical vapor deposition (CVD) diamond. CVD diamond can be classified as either single crystal or polycrystalline. Either type can be manufactured to produce materials that range from opaque to fully transparent. Typical impurities within CVD diamond are graphite and hydrogen, although trace amounts of other materials may be present, such as nitrogen. In addition to impurities, there are structural defects which occur that further degrade the material and its properties as compared to a defect free natural diamond. As a result, CVD diamond is often opaque or very dark

Most industrial applications for diamond require high quality crystals or films. Common applications include lenses that require high optical transmission of light, heat sinks that require very high heat conductivity, and electrical insulators. Prior work to improve these materials by high temperature treatment has shown that heating above 850° C. significantly degrades the sample. In fact, temperatures above 1600° C. have totally destroyed sample integrity due to formation of cracks thought to be the result of loss of bonded hydrogen or conversion of the diamond carbon to graphite.

Natural or synthetic diamond, on the other hand, can withstand treatment to very high temperatures. In fact, it has been shown that annealing of synthetic and natural type I or type II diamonds in the range of 1900° C. to 2600° C. at pressures in the range of 50 to 80 kbars causes the visible color of the diamond to change. In the case of natural diamond type I, the color changes from brown to yellow or yellow-green. For type II natural diamond, the color changes from brown to colorless or, on rare occasions, blue or pink. Synthetic diamond will change from yellow to lighter yellow.

It would be advantageous if a method were devised that would significantly improve the properties of CVD diamond after it is grown. It would also be desirable to form CVD diamond with fewer defects that serve to degrade the intrinsic properties of a perfect crystalline diamond material in order to enhance its usage in many applications.

SUMMARY

According to the present invention, there is provided a method of improving the optical properties of CVD diamond, which includes the steps of:

1. creating a reaction mass by placing the CVD diamond in a pressure transmitting medium that completely encloses the diamond; and

2. subjecting the reaction mass to a temperature of at least 1500° C. and, preferably, in the range of about 1800° C. to about 2900° C. under a pressure of at least 4.0 GPA.

The period of time during which the sample is subjected to HPHT conditions is from less than about one minute to about 30 minutes. The preferred time is between one to five minutes. The actual conditions can be varied depending on the grade and the size of the CVD sample.

The reaction mass may be subjected to any number of such treatments. Thus, if the desired results were not achieved the first time, the sample may be re-treated at HPHT until such time that the desired improvement in characteristics or properties is achieved.

The greatest improvements will be noted for single crystal CVD diamond that is void of defects, such as surface pits, microscopic inclusions, and that is at least partially translucent. Such CVD material may be so improved as to even be polished and faceted to produce a gem quality diamond to be used in jewelry.

BRIEF DESCRIPTION OF THE DRAWING

The various features, advantages and other uses of the present invention will be come more apparent by referring to the following detailed description and drawing in which:

FIG. 1 is a cross sectional view of the inner portion of a high pressure cell used to treat CVD diamond according to the present invention.

DETAILED DESCRIPTION

The present invention is a process to heat treat single crystal CVD diamond at high temperature and high pressure. It is not certain what happens to the CVD material when it is subjected to such conditions. Possibly, internal atoms shift position to more correctly align themselves to the diamond crystalline structure or perhaps the bonding mechanism shifts such that SP^2 type bonds become SP^3 type bonds causing carbon atoms to change from impurity status to becoming part of the diamond crystal lattice.

Whatever the mechanism, it has been found that treating CVD diamond at high pressure and high temperature (HPHT) causes the optical properties to change so much that opaque material become clear. This same mechanism also improves the thermal conductivity and the electrical resistance for the CVD diamond. This is very unexpected since prior work has shown just the opposite occurs when CVD diamond is annealed in a vacuum to 1600° C. (A. V. Khomich et al., Diam. Relat. Mater. 10 (2001), pp. 546–551), Heating CVD diamond in vacuum has caused diamond to darken at temperatures as low as 850° C. (S. Mitra, K. I. Gleason, Diam. Relat. Mater. 2 (1993) p. 126).

Thus, one would expect that when diamond is heated to temperatures above 850° C., at pressures where graphite is the stable phase, significant degradation of the sample would result. However, very unexpected behavior occurs in CVD diamond at high temperatures where the pressure is raised above atmospheric pressure but still remains within the graphite stable region. Under certain conditions of temperature and pressure, CVD diamond does not degrade; instead the opposite occurs: the sample is transformed into a more perfect diamond crystalline material

FIG. 1 shows a cross section of the inner portion of an assembly that may be employed to treat CVD diamond 1 according to the present invention. The outer body 2 is cylindrical in shape and is designed to fit within a central cavity of an ultrahigh pressure and ultrahigh temperature cell, such as that described in U.S. Pat. Nos. 3,745,623 or 3,913,280.

The outer body 2 is composed of graphite or other material that will readily transmit pressure and remain stable and non-reactive to the CVD diamond 1 at high temperature and high pressure. Other materials for the outer body 2 include, but are not limited to, salt, MgO, or talc. The CVD diamond 1 is encapsulated in the outer body 2. The CVD sample 1 is a stand alone CVD diamond or a CVD coating on diamond or other materials. This assembly should be consolidated to greater than 90% of its theoretical density and made to fit snugly into a HPHT reaction cell, such as that used to manufacture PCD.

The entire cell is subjected to pressures in excess of 4.0 GPA and heated to temperature in excess of 1500° C. for a time of five minutes. Then the cell is allowed to cool enough so that the CVD diamond does not back-convert to graphite after the pressure is released.

After pressing, the sample **1** is removed from the graphite outer body by mechanical means, such as by tapping with a mallet. The sample can then be further heated in an oven to 725° C. for approximately ten minutes in order to obtain a clean and smooth outer diamond surface. This treatment removes any graphite that may have adhered to the sample. The surface can also be polished in a manner as traditionally used on natural diamond single crystals or polycrystalline diamond compacts.

EXAMPLE #1

A cubed-shaped CVD coated synthetic type Ib diamond, approximately one centimeter square, was encapsulated in a graphite cylinder. The CVD portion was a layer on one side of the cube-shaped natural diamond, approximately one millimeter thick and was opaque to optical transmission. The synthetic diamond substrate was light yellow.

The graphite cylinder was loaded into a HPHT reaction vessel that was configured for indirect heating of the reaction mass. Various reaction vessel configurations, which provide the indirect or direct heating, are disclosed in the patent literature and are also useful for carrying out the present HPHT process.

Reaction vessels of this type usually of a plurality of interfitting cylindrical members and end plugs or discs for containing a sample in the innermost cylinder. For the indirectly heated type of reaction vessel, one of the cylindrical members is made of graphite that is heated by the passage of electric current through the cylinder. For this case, the reaction mass, if composed of graphite, must be electrically insulated from the graphite heater tube by an insulating material, such as talc or salt, to prevent passage of electrical current through the reaction mass. In the directly heated type of reaction vessel, the insulating sleeve is not required as the sample is heated by simply passing electric current through the reaction mass provided it is composed of an electrically conducting material, such as used in this example.

The reaction vessel was placed in a conventional HPHT apparatus. First, the pressure was increased to 5.0 GPA, and then the temperature was rapidly brought up to 2200° C. The sample was maintained at these conditions for five minutes, then the temperature was decreased over a period of about one minute to room temperature before the pressure was released.

The sample was removed from the reaction mass and examined under an optical microscope. The opaque CVD diamond layer turned clear and remained firmly bonded to the yellow synthetic type Ib diamond.

What is claimed is:

1. A method to improve the optical clarity of CVD diamond where the CVD diamond is single crystal CVD diamond, by raising the CVD diamond to a set temperature of at least 1500° C. and a pressure of at least 4.0 GPA outside of the diamond stable phase.

2. The method of claim **1** wherein the CVD diamond is a single crystal coating upon another material.

3. The method of claim **1** wherein the step of raising the temperature of the single crystal CVD diamond further comprises the step of:

raising the single crystal CVD diamond to a set temperature of about 1800° C. to about 2900° C.

4. The method of claim **1** wherein the step of raising the temperature of the single crystal CVD diamond further comprises the step of:

maintaining the temperature of the single crystal CVD diamond at the set temperature for less than about one minute.

5. The method of claim **1** wherein the step of raising the temperature of the single crystal CVD diamond further comprises the step of:

raising the temperature of the single crystal CVD diamond to at least 1500° C. over a time period of about one minute to five minutes.

6. The method of claim **1** wherein the step of raising the temperature of the single crystal CVD diamond comprises of the step of:

raising the temperature of the single crystal CVD diamond to about 2200° C. at a pressure of about 5.0 GPA.

7. The method of claim **1** further comprising the step of:

after reaching the set temperature, decreasing the temperature of the CVD diamond to ambient temperature while maintaining the pressure on the single crystal CVD diamond.

* * * * *

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APPLICATION NUMBER	FILING or 371(c) DATE	GRP ART UNIT	FIL FEE REC'D	ATTY.DOCKET.NO	TOT CLAIMS	IND CLAIMS
12/362,529	01/30/2009	1792	1650	056100-5078	7	1

CONFIRMATION NO. 1281

9629
MORGAN LEWIS & BOCKIUS LLP
1111 PENNSYLVANIA AVENUE NW
WASHINGTON, DC 20004

UPDATED FILING RECEIPT

OC000000035244637

Date Mailed: 03/30/2009

Receipt is acknowledged of this reissue patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Applicant(s)

Wei Li, Ann Arbor, MA;
Russell J. Hemley, Washington, DC;
Ho-kwang Mao, Washington, DC;
Chih-shiue Yan, Washington, DC;

Assignment For Published Patent Application

CARNEGIE INSTITUTION OF WASHINGTON, Washington, DC

Power of Attorney: The patent practitioners associated with Customer Number 09629

Domestic Priority data as claimed by applicant

This application is a REI of 10/161,266 06/03/2002 PAT 6,811,610

Foreign Applications

If Required, Foreign Filing License Granted: 02/24/2009

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 12/362,529**

Projected Publication Date: None, application is not eligible for pre-grant publication

Non-Publication Request: No

Early Publication Request: No

Title

Method of Making Enhanced CVD Diamond

Preliminary Class

427

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at http://www.uspto.gov/web/offices/pac/doc/general/index.html.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, http://www.stopfakes.gov. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4158).

LICENSE FOR FOREIGN FILING UNDER

Title 35, United States Code, Section 184

Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as

set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign AssetsControl, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

UNITED STATES PATENT AND TRADEMARK OFFICE

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APPLICATION NO.	ISSUE DATE	PATENT NO.	ATTORNEY DOCKET NO.	CONFIRMATION NO.
12/362,529	04/06/2010	RE41189	056100-5078	1281

9629 7590 03/17/2010

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ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

Determination of Patent Term Extension or Adjustment under 35 U.S.C. 154 (b)

A reissue patent is for "the unexpired part of the term of the original patent." See 35 U.S.C. 251. Accordingly, the above-identified reissue application is not eligible for Patent Term Extension or Adjustment under 35 U.S.C. 154(b).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Data Management (ODM) at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site http://pair.uspto.gov for additional applicants):

Wei Li, Ann Arbor, MA;
Russell J. Hemley, Washington, DC;
Ho-kwang Mao, Washington, DC;
Chih-shiue Yan, Washington, DC;